70503

HUDSON RIVER FISH/SEDIMENT PCB ANALYSIS PROJECT

New York State Department of Environmental Conservation

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Project Quality Assurance Officer: Robert W. Bauer
Robert W. Bauer

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322824

1. Project Name : Hudson River Fish/Sediment PCB Analysis Project.

2. Project Requested By: Jay Field, National Oceanic and Atmospheric Administration, Seattle, Washington

3. Date of Request: September 1992.

4. Date of Project Initiation: Summer 1993

5. Project Officer: Ronald J. Sloan

6. Quality Assurance Officer: Robert W. Bauer

7. Project Description:

A. Background

On September 25, 1984 the United States Environmental Protection Agency (EPA) signed the Record of Decision (ROD) regarding the PCB contamination of the Hudson River. The ROD found that cost-effective technology was not available to mitigate the damage to public health and the environment by the PCB contamination of the riverbed (NYSDEC 1989). Concerns raised over health and fisheries use issues led the New York State Department of Environmental Conservation to develop a case in 1989 for reconsideration of the ROD.

In late 1989 the United States Environmental Protection Agency announced that the 1984 Hudson River sediment NO ACTION decision would be reassessed. As part of the reassessment process, an Ecological Risk Assessment(ERA) was included in the Phase 2 Work Plan. The ERA plan, however, does not include any further sampling of biota, but it does provide for sediment sampling from 15 locations throughout the course of the river from control areas near rivermile(RM) 210 to rivermile 24 at Piermont Marsh. Of all the reports and publications reviewed and listed in the recent Phase 1 Report on the Hudson River PCB Reassessment (TAMS and Gradient 1991), none attempted to develop a direct linkage between PCB in sediment deposits and concentrations in biota. The proposed plan, the HUDSON RIVER FISH/SEDIMENT PCB ANALYSIS PROJECT, is an attempt to fill this data void.

Therefore, the goal is:

> To provide a basis for an ecological risk assessment pertaining to PCB in the Hudson River that will link PCB in sediment to concentrations in the biota. This would form a basis for evaluating the efficacy of remediation and provide a framework for long-term monitoring of the system following remediation.

However, with the increased comprehension over the magnitude of PCB still emanating from the Baker Falls area, there is the potential masking of the specific influence/importance of sediment PCB concentrations on fish PCB levels. Hence, this plan, compared to earlier drafts, provides for some mechanism(s) to alleviate partially confounding influences on eventual data interpretation.

B. Objectives (dual):

The objectives are: A) to sample at least 19 stations along the Hudson River for concentrations of PCB, principally on a congeneric basis, in sediment, benthic invertebrates, and localized species of fish during the ecological risk assessment portion of the Phase 2 Hudson River PCB Reassessment; and B) to sample ı0 reaches for mobile fish species during the same interval to ascertain PCB conditions over a broader area than that achieved in Objective A.

C. Data usage:

The data will be used to describe relationships between sediment, invertebrates and fish PCB concentraions as functions of congener and homolog composition, nature of sediment (e.g. organic carbon content) and distance from a dominant PCB source (e.g. Ft. Edward and Hudson Falls area). They will also provide basic information to select key locations to evaluate remedial efforts and to establish a long-term, cost-effective monitoring design for PCB in the Hudson River.

D. Sampling Network Design and Rationale:

Fish act as integrators of chemical contaminant inputs to their ecosystem and serve as indicators of the severity of contamination in the biotic community. Fish are also important for socioeconomic reasons and often provide much of the impetus for cleanup of contaminated environments. Contaminated sediment may function as source(s) of the contaminant that accumulates in the

biotic components.

Ideally, sampling of organisms residing directly over, or in close juxtaposition to a contaminant source would provide the most sensitive test for describing biota/sediment PCB relationships. In natural environments, however, this is not always possible. Therefore, to maximize the potential for exposure to localized conditions, relatively sedentary species with minimal home ranges, at least during the season of collection, are sought. More mobile, larger species with larger home ranges may be more effective integrators of contamination over broader areas. Hence, there are two basic types of fish that are involved with the envisioned sampling effort:

A. Localized, sedentary, minimal home range during the season of collection. Possible species groups - minnows, darters, sunfish, killifish.

B. Mobile, wide home range but not anadromous that function as effective integrators of contamination over a broader area than those species which may reflect more local influences. Possible species - carp, white perch, yellow perch, brown bullhead, white catfish. Collections for mobile species are targeted toward yellow perch above the Federal Dam at Troy and white perch south of Troy.

For group A (localized) fish, sampling at any one location should consist of as many species as possible to comprise three whole fish composites of ten fish each for each species. The purpose for the extensive collections at a given site is to maximize the likelihood that one species will be represented across all sampling locations. In the likely event that a common species will not occur, additional species will provide for overlapping species comparisons so that continuity in the biotic contamination gradient is maintained. Excess specimens will be retained in the event additional analyses are warranted or independent investigators have use for them.

For group B (mobile) species, the same compositing strategy is employed, except if the fish collected are exceptionally large (i.e. >305mm total length) or widely divergent in sizes (i.e. >100% difference in body weight or total length between the smallest and largest specimens of the same species). In such cases the number of fish in each composite may be reduced to three fish per composite but the number of analyses is increased to five. If ictalurids are selected

for analysis, due to analytical preparation problems, they are ground and homogenized without skins. Other species are prepared whole. Fish collected should be of adult sizes. As in group A, excess specimens will be retained until the study is completed.

Note: The selection of species for analysis will occur after the collections are completed. This will maximize the likelihood of having one species represented across as many sampling areas as possible. Where it is not possible to have a common species represented, additional species will be retained. Some locations will, of necessity, be represented by more than one species, within the 'A' and 'B' designations, to provide continuity through the spatial gradient of the river.

A summary of the sampling design for both groups of fish is provided in Table 1.

Collections are slated to commence on August 1, 1993 in conjunction with sediment sampling and collections of benthic invertebrates from several locations. Protocols for sediment and invertebrate sampling are presented in a separate document prepared by TAMS Consultants, Inc. under contract by USEPA as part of the Hudson River Reassessment. Fish are collected according to standard procedures outlined in Appendix I. These procedures include provisions for maintaining field collection records and chain of custody requirements.

Sampling locations for fish and sediment are tentatively targeted as listed below. Final and specific sites are dependent upon field verifications. Selected locations will be photographed, described and plotted on 7.5 minute topographic maps.

Suggested Hudson River Ecological Sampling Stations

RM212(approx.) Probable background sample, since it is above the direct influence of the Spier Falls facility. Group A fish species are targeted, which will augment another collection from the spring of 1992 of Group B types. Retain Group B species, however, during the August collection. Possibility of a control benthic invertebrate collection involved at this location.

RM210(approx.) No longer considered "background" due to finding of a small PCB disposal site above

Table 1. Sampling design for linkage of PCB concentrations between sediment and fish. Projected for fish only.

Fish Type	Number of Stations	Projected Overlap Stations	# composite Samples/ Location	Total Number
A. localized	19	4	3	69
B. mobile	10	3	5(max.) 3(min.)	65(max.) 39(min.)
	TOTALS	-maximum = -minimum =		134 108

the Sherman Island dam. Execute sampling for <u>two</u> locations within this pool between the dams. One location should include the reference disposal area. The other location will be above the waste site but below the Spier Falls dam. Groups A & B fish species.

RM203 This area is reflective of baseline conditions for the upper river. Some PCB is present in the fish, presumably due to upstream influences. High resolution (HR) coring RM203 (TAMS). Groups A & B fish species.

RM196.8 Above the remnant area but below Bakers Falls. High concentrations in water during 1991 and 1992 determined by General Electric Company in their monitoring activities associated with the remnant capping. Group A fish species.

RM194 At Rogers Island which is below the remnant deposits and the Hudson Falls discharge point and in the vicinity of the discharge from the Ft. Edward plant. HR coring RM195 (TAMS). Groups A & B fish species.

RM189 Above Thompson Island Dam. HR coring RM189 (TAMS). Groups A & B fish species.

<u>Two</u> locations within this area will involve fish sampling. One is at RM 189.5 (Species A & B), which is on western bank at south end of Griffin Island. This is an established trend species sampling location. The second location will most likely be at RM191.5, eastern bank across river from the mouth of the Snook Kill. 'A' type species only. These locations will also involve invertebrate sampling, but three additional invertebrate locations are envisioned for the Thompson Island Pool: RM189 - west side of jetty on east side of river which leads into the navigation channel; RM188.5 - west bank just above Thompson Island Dam; RM190.5 - east bank just south of McDonald Oil Company.

RM175 Stillwater Pool is a fish collection location area. Weedbeds 1.5 miles upstream from the Stillwater Dam on the east shore appears to be a good depositional area and is close to HR coring locations of TAMS RM166 and RM168. Groups A & B fish species.

RM157 Immediately below Lock 1 dam, but well above confluence with the Mohawk River. Major fish collections occurred in 1991 and 1992 between Lock 1 and the Federal Dam. HR coring RM158 (TAMS). Group A fish species.

RM142 Yearling pumpkinseed collection location in the historical Port of Albany south turning basin but may not represent a good depositional area, although a TAMS HR coring RM143 is indicated in the vicinity. Group A fish species.

RM135 Below Shad Island at lower end of Binnen Kill, a tidal creek, access to shore and depositional areas may be possible. Group A fish species.

RM123 Little Nutten Hook area of Stockport Flats reputed to have bald eagle nesting in the vicinity. Group A fish species. Benthic invertebrate sampling will also occur.

RM115-118 Rogers Island area on west side could be good depositional and sampling area. Fish collections of more mobile species have come from mouth of Catskill Creek and Ramshorn Marsh. Groups A & B fish species.

RM100 Tivoli Bays are restricted from mainstem flows. Sediment sampling not appropriate within the bays.Sample outside trestles in main river near Magdalen Island and Cruger Island. Group A fish species. Benthic invertebrate sampling will also occur.

RM88 Esopus Meadows just north of Esopus Village and across river from Vanderburgh Cove. Fish collections have occurred here. HR coring RM91.8 (TAMS). Groups A & B fish species. Benthic invertebrate sampling will also occur.

RM58 Stay north of Moodna Creek and use north side of Plum Point. HR coring RM60 (TAMS). Groups A & B fish species.

RM40 Iona Island (probably in marsh on south side of the island) - stay on the riverside of the railroad bed and in shallows along the riverbank. Group A fish species. Benthic invertebrate sampling will also occur.

RM24 Piermont Marsh - stay to southern end to minimize

influence of the Spar Kill. Groups A & B fish species. Benthic invertebrate sampling will also occur.

Considerations for site selections:

-- should reflect mainstem conditions not those at the mouths of tributary streams or resulting from local runoff.

-- should be locations where sedimentation occurs and contains sufficient amounts of fines and organic material to ensure relatively high concentrations if deposition does occur.

-- effect of secondary sources in tributaries would tend to dilute rather than increase PCB concentrations in the Hudson River.

-- large areas along the river and the canal are dredge spoils. Sampling of these may cause erroneous results and interpretations. Presumably information on these areas are available through NYSDOT, now the Thruway Authority, for the upper river and in the lower river from the Army Corps of Engineers.

E. Montitoring Parameters and Frequency of Collection

This monitoring plan is envisioned as a one-time event. However, if results suggest and conditions warrant, elements of this project may be modified as part of the evaluation of the effectiveness of remediation and may become incorporated into the long-term trend analysis for PCB in the Hudson River. Parameters to be examined and recorded in the field are species, location, collection date, collection method, collection agents, fish lengths and weights. USEPA's contract laboratory, Aquatec, Inc. located in Colchester, VT, will determine lipid content and wet weight PCB concentrations at the congeneric level. The DEC Hale Creek Field Station Analytical Services Laboratory will determine and record lipid content, PCB (total, and various "Aroclors"), and several organochlorine pesticides for limited numbers of samples in addition to selected split samples of ground and homogenized tissue prepared by Aquatec. Lipid content may be more critical in interpreting results than trophic status.

8. Project Fiscal Information

The budget for this project (fish collections only) appears in Table 2. Analytical costs are not included in this plan, because they were negotiated with Aqua-Tech by USEPA. Since the sampling locations above Bakers Falls involve another hazardous waste site, fish analytical costs associated with RMs 212, 210 and 203 may be assumed by the Niagara Mohawk Power Corporation.

9. Schedule of Tasks and Products

Sampling	August 1 - Sept. 15, 1993
Transportation	September 1993
Chemical analyses	September - November 1993
Reporting	December 1993 (raw data and summary reports only)

10. Project Organization and Responsibility

The following is a listing of key project personnel and their responsibilities:

Helen Chernoff (TAMS Consultants)	Coordination of sediment and benthic invertebrate collections (separate plan), sample storage and transportation and sample QC.
Les Saltsman (NYSDEC, Region 5)	Coordination of and cooperation in collection efforts occurring above the Federal Dam at Troy.
Kathy Hattala (Hudson R. Unit)	Coordination of and cooperation in collection efforts below the Federal Dam at Troy.
Samuel Jackling (NYSDEC)	Sample preparation, laboratory analysis, laboratory data management and QC on selected samples.
Aquatec, Inc.	Sample preparation, laboratory analysis, laboratory data management and QC on congeneric PCB evaluations.
Robert Bauer (NYSDEC)	Performance and system auditing; overall quality assurance.
Ronald Sloan	Overall project coordination, data management and reporting.
NMPC	Niagara Mohawk Power Corporation and their contractors. Possible assistance

Table 2.

BUDGET ESTIMATE FOR FY '93-'94 OF THE LINKAGE OF HUDSON RIVER FISH WITH SEDIMENT PCB ANALYSIS PROJECT - ALL STUDY SEGMENTS COMBINED INCLUDING:

COLLECTIONS ONLY - DEC INVOLVEMENT

LOCALIZED AND MOBILE SPECIES

		Staff days	Amount
A.	Sampling, processing and transportation		
	1a. Personnel		
	Conservation biologists	20	$ 3,040
	Research Scientist	20	4,340
	Technicians	80	7,200
	Subtotal Oversight		14,580
	1b. Fringe benefits (30.04% of personnel costs)		4,380
	Subtotal personnel & fringe		18,960
	2. Supplies and materials		2,500
	3. Travel		1,500
	Subtotal s&m travel		4,000
	Total Sampling		22,960
B.	Project oversight, data management and reporting		
	Research Scientist	60	13,020
	QA Officer	3	588
	Supervising Ecologist	3	651
	Keyboard Specialist	3	243
	Subtotal oversight		14,502
	Fringe Benefits (30.04% of personnel)		4,356
	Total Project oversight		18,858
C.	Indirect costs (34% of Department personnel costs)		12,858
	TOTAL (A + B + C)		**$54,676**

in collections above Glens Falls and in meeting analytical costs in that reach.

11. Data Quality Requirements and Assessments

Detection and quantitation limits, estimates of accuracy and precision are provided for each chemical parameter in the "Standard Operating Procedures Manual" for the Analytical Services Laboratory at the Hale Creek Field Station, NYSDEC. Any other laboratory participating in this project will follow similar procedures. A synopsis of these needs are included herein as Appendix II.

12. Sampling Procedures

Sampling will utilize standard collecting methods including gillnetting, seining, trapnetting, trawling, electroshocking and angling. All samples are subject to standard handling and recording procedures as outlined in Appendix I. A copy of the data is sent to the project coordinator.

13. Sample Custody Procedure

The completed "Fish/Wildlife Continuity of Evidence" (Appendix I) form must accompany the samples. A copy of the final form is sent to the project coordinator.

14. Calibration Procedures and Preventive Maintenance

A. Calibration procedures - All instrumentation is calibrated daily. Additional standards are run periodically during the day to verify continued instrument calibration.

B. Preventive Maintenance
   1. HP5890 Series II and HP5880A GC's at Hale Creek Field Station
      a. Septa and glass wool changed weekly.
      b. Gas filters/purifiers changed every 6 months.
      c. Columns are changed when needed, usually when resolution or response of standards run change by 30%. This is a subjective opinion.
   2. Contract laboratories are expected to pursue similar courses of action.

15. Documentation, Data Reduction, and Reporting

A. Documentation: All data from gas chromatographs (GC) are generated on hard copy and disk and stored in files at the laboratories. Results are compiled and sent to R. Sloan for review and reporting. Error checks involve 10% of all data from start to finish. Compiled data from the field for geographic and biological information and from the analytical laboratory for chemical results must conform for each variable as per the standardized data dictionary developed jointly between the Division of Fish and Wildlife (Bureaus of Environmental Protection and Wildlife) and the New York State Department of Health. The data dictionary was developed to ensure compatability between scientific/technical needs, environmental and public health concerns, and Geographic Information Systems (GIS) practices. A description of the data dictionary appears as Appendix III. Modifications to the dictionary are made as appropriate to reflect changes or improvements in analytical methodology or as project needs dictate.

B. Data Reduction and Reporting: Data are compiled in tables, subjected to statistical analyses where appropriate and reported with explanatory text.

16. QA/QC Data Review

Data from the blanks, spike recoveries and duplicate analyses are reviewed by Samuel Jackling and other chemists conducting analyses. A minimum of 10% of all data are recalculated by the respective participating laboratories. Overall data are reviewed by R. Sloan.

17. Performance and Systems Audits

Laboratories are expected to participate or have participated in several performance evaluation studies such as EPA Water Pollution Performance studies, International Joint Commission Great Lakes Monitoring Program, and NYSDEC Interlaboratory studies. Standard reference materials for biological samples are available for performance checks through NIST.

18. Corrective Action

When a QC sample falls outside the control limits, the QC sample is rerun. If the QC sample is still outside the control limits, that day's results for all samples are voided, analytical problem is identified and corrected, and the samples rerun.

19. Reports

The findings from this project will be reported to parties interested in or having jurisdiction over issues associated with the Hudson River. Raw and summary data will be turned over to the New York State Department of Health for health concern considerations as appropriate by January 1993.

LITERATURE CITED

NYSDEC. 1989. The 1984 Superfund Decision for the Hudson River: The Case for Reconsideration. New York State Department of Environmental Conservation, Albany, New York. August 25, 1989. 10 pp.

TAMS and Gradient. 1991. Phase 1 Report Interim Characterization and Evaluation Hudson River PCB Reassessment RI/FS. EPA Work Assignment No. 013-2N84. TAMS Consultants,Inc. and Gradient Corporation.

APPENDIX I

Fish Collection and Preparation Procedures

GENERAL NEW YORK STATE

FISH COLLECTION PROCEDURES

A. Following data are to be taken on each fish collected:

1. Date collected

2. Species identification (please be explicit enough to enable assigning genus and species)

3. Total length (nearest mm or smallest sub-unit on measuring instrument) and weight (nearest g or smallest sub-unit of weight on weighing instrument). Take all measures as soon as possible with calibrated, protected instruments (e.g. from wind and upsets) and prior to freezing.

4. Method of collection (gill net, hook and line, etc.)

5. Sample location (Waterway and nearest prominent identifiable landmark).

6. Sex - fish may be cut enough to allow sexing, but do not eviscerate.

7. Tag number (each specimen to be individually tagged with jaw tag).

Record length and weight as soon as possible after collection and before freezing. Other data are recorded in the field upon collection. An age determination of each fish is optional, but if done, it is recorded in the appropriate "Age" column.

The original of all collection record and continuity of evidence forms shall accompany delivery of fish to the lab. A copy shall be directed to Larry Skinner or Ron Sloan. All necessary forms will be supplied by the Bureau of Environmental Protection.

Please submit photocopies of topographic maps or good quality navigation charts indicating sampling locations. These records are of immense help to us (and hopefully you) in providing documented location records which are not dependent on memory and/or the same collection crew. In addition, they may be helpful for contaminant source trackdown and control efforts of the Department.

B. Each fish to be wrapped in a plastic bag. The Bureau of Environmental Protection will supply the bags.

C. Groups of fish, by species, to be placed in one large plastic bag per sampling location. The Bureau of Environmental Protection will supply the larger bags.

D. Do not eviscerate.

E. All fish must be kept at a temperature below 45°F immediately following data processing. As soon as possible, freeze at 0°F ± 10°F. Due to occasional freezer failures, daily freezer temperature logs are required.

F. Prior to any delivery of fish, coordinate delivery with, and send copies of the collection records, continuity of evidence forms, and freezer temperature logs, to:

Larry Skinner or Ron Sloan
Bureau of Environmental Protection
Room 530
50 Wolf Road
Albany, New York 12233-4756
Telephone: (518) 457-1769

Samples will then be directed to:

The analytical facility and personnel noted on specific project descriptions.

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
BUREAU OF ENVIRONMENTAL PROTECTION

FISH PREPARATION PROCEDURES FOR CONTAMINANT ANALYSIS

Background

New York State Department of Environmental Conservation (DEC) conducts studies requiring chemical analysis on fish tissues. Routine monitoring and surveillance studies develop data on contaminants in fish for several reasons:

1. To identify sources of environmental contamination;

2. To identify the geographic extent of environmental contamination;

3. To identify temporal trends of contaminants in fish and wildlife; and

4. To provide information regarding human consumption advisories.

Chemical analyses of edible fish flesh have been determined to be the most appropriate analyses for satisfying all of these objectives. The following methodology has been developed in order to standardize the tissues under analysis and to adequately represent the contaminant levels of fish flesh. The methodology is slightly modified from the U.S. Food and Drug Administration procedures. The portion of edible flesh analyzed will be referred to as the standard fillet unless otherwise noted. For some species, the procedure is modified as indicated below.

Procedures for Standard Filleting

1. Remove scales from fish. Do not remove the skin.

2. Make a cut along the ventral midline of the fish from the vent to the base of the jaw.

3. Make diagonal cut from base of cranium following just behind gill to the ventral side just behind pectoral fin.

4. Remove the flesh and ribcage from one-half of the fish by cutting from the cranium along the spine and dorsal rays to the caudal fin. The ribs should remain on the fillet.

5. Score the skin and homogenize the entire fillet.

Modifications to Standard Fillet

Four modifications of the standard fillet procedure are designed to account for variations in fish size or known preferred preparation methods of the fish for human consumption.

1. Some fish are too small to fillet by the above procedure. Fish less than approximately 6 inches long and rainbow smelt are prepared by cutting the head off from behind the pectoral fin and eviscerating the fish. Ensure that the belly flap is retained on the carcass to be analyzed. When this modification is used, it should be noted when reporting analytical results.

2. Some species are generally eaten by skinning the fish. The skin from these species is also relatively difficult to homogenize in the sample. Hence, for the following list of species, the fish is first skinned prior to filleting:

Brown bullhead	White catfish
Yellow bullhead	Channel catfish
Atlantic sturgeon	Lake sturgeon
Black bullhead	

3. American eel are analyzed by removing the head, skin, and viscera; filleting is not attempted.

4. Forage fish and young-of-year fish are analyzed whole. This category is considered to be less than 150mm (6 inches).

C H A I N O F C U S T O D Y*

NEW YORK STATE
DEPARTMENT OF ENVIRONMENTAL CONSERVATION

I, _____, of _____ have collected the
(Print Name) (Print Address)
following on _____, 198__ from _____ in the
vicinity of _____ Town of _____,
_____ County.

Item(s): _____

_____

_____

said sample(s) were in my possession and handled according to standard procedures provided to me prior to collection. The sample(s) were placed in the custody of a representative of the New York State Department of Environmental Conservation on _____, 198__.

_____ _____
Signature Date

I, _____, have received the above mentioned sample(s) on the date specified and have assigned identification number(s) _____ _____ to the sample(s). I have recorded pertinent data for the sample(s) on the attached collection records. The sample(s) remained in my custody until subsequently transferred, prepared or shipped at times and dates as attested to below.

_____ _____
Signature Date

SECOND RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
THIRD RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
FOURTH RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
RECEIVED IN LABORATORY BY (Print Name)		TIME & DATE
SIGNATURE	UNIT	
LOGGED IN BY (Print Name)	TIME & DATE	ACCESSION NUMBERS:
SIGNATURE	UNIT	

*See Reverse Side.

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NOTICE OF WARRANTY

By signature to the chain of custody (reverse), the signator warrants that the information provided is truthful and accurate to the best of his/her ability. The signator affirms that he/she is willing to testify to those facts provided and the circumstances surrounding same. Nothing in this warranty or chain of custody negates responsibility nor liability of the signators for the truthfulness and accuracy of the statements provided.

HANDLING INSTRUCTIONS

On day of collection, collector(s) name(s), address(es), date, geographic location of capture (attach a copy of topographic map or navigation chart), species, number kept of each species, and description of capture vicinity (proper noun, if possible) along with name of Town and County must be indicated on reverse.

Retain organisms in manila tagged plastic bags to avoid mixing capture locations. Note appropriate information on each bag tag.

Keep samples as cool as possible. Put on ice if fish cannot be frozen within 12 hours. If fish are held more than 24 hours without freezing, they will not be retained or analyzed.

Initial recipient (either DEC or designated agent) of samples from collector(s) is responsible for obtaining and recording information on the collection record forms which will accompany the chain of custody. This person will seal the container using packing tape and writing his signature, time and date across the tape onto the container with indelible marker. Any time a seal is broken, for whatever purpose, the incident must be recorded on the Chain of Custody (reason, time and date) in the purpose of transfer block container then is resealed using new tape and rewriting signature, with time and date.

82-14-6[illegible](84)

FISH/WILDLIFE COLLEC[illegible]ON RECORD
NEW YORK STATE DEPARTMENT OF EN[illegible]NMENTAL CONSERVATION
DIVISION OF FISH AND WILDLIFE

FROM REGION_____FOR_____TOXIC SUBSTANCE MONITORING PROGRAM

BY COLLECTOR(S)_____USING_____COLLECTION METHOD.

SPECIMENS PRESERVED BY_____METHOD.

FILL IN APPROPRIATE BLANKS AS COMPLETELY AS POSSIBLE.

FOR LAB USE ONLY LAB ENTRY NO.	COLLECTION OR TAG NO.	SPECIES	DATE TAKEN	LOCATION	AGE	SEX &/OR REPROD. CONDIT.	LENGTH ( )	WEIGHT ( )	REMARKS

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APPENDIX II

Analytical and Laboratory Procedures

at

Hale Creek Field Station

DETERMINATION OF MERCURY IN TISSUES
NYS DEPARTMENT OF ENVIRONMENTAL CONSERVATION
Hale Creek Field Station
Toxic Substances Monitoring Laboratory

Reference: Analytical Chemistry 40, 2085, 1968

1. DIGESTION:

Weigh portions of tissue samples containing up to 1g of wet tissue into 300 mL G-S Erlenmeyer flasks for analysis. (Sample sizes from known high Hg areas can be adjusted so as to contain ca 1 ug Hg. Samples larger than ca 1g wet or 0.25g freeze dried tissue may be difficult to digest.) Add 25 mL sulfuric acid to each flask. Insert packed air condenser into each flask and place in 70 C water bath (using lead donuts to stabilize). Allow ca 30 min for acid to char samples. Remove flasks from water bath and partially remove air condensers one at a time. Add 3 mL of 30% hydrogen peroxide to each flask. Replace air condensers, swirl to mix and return flasks to water bath for ca 2 hr. Swirl flasks to mix contents one or two times during digestion. Swirling acid can be used to dislodge sample particles from walls of flask above normal liquid level. After addition of hydrogen perioxide, solns should fade from brown to yellowish to clear. Some yellow coloration may return as digestion progresses. Remove flasks from water bath at end of 2 hr. digestions and place in ice bath. Add 95 mL distilled water to each flask through air condenser to recover any mercury which may have been volatilized and condensed during the digestion. Cool in ice bath to near room temperature. Remove flasks from ice bath and remove air condensers. Add 4 mL of 5% potassium permanganate to each flask. Rinse down inside walls of flask with ca 5 mL of water. Stopper flasks.

2. STANDARDS:

Standard solns are prepared daily by dilution of 1000 ug/mL stock soln. (Fisher certified reference Solution SM114-500, or equivalent.)

Diln #1: 10 mL of stock diluted to 1 l with 0.25n sulfuric acid (contains 10 ug/mL)

Diln #2: 25 mL of diln #1 diluted to 250 mL with 0.25 n sulfuric acid (contains 1 ug/mL)

Diln #3: 25 mL of diln #2 diluted to 500 mL with 0.25 n sulfuric acid (contains 1 ug/20 mL)

Working standards are prepared to contain the same acid and potassium permanganate contents and volume as the digested samples, from diln #3 as follows:

ug/Hg	mL Std Diln #3	mL H2O	mL 5% KMnO4	mL 1+2 H2SO4
0.25	5	45	4	75
0.5	10	40	4	75
1.0	20	30	4	75
1.5	30	20	4	75
2.0	40	10	4	75
2.5	50	0	4	75

Generally, only the 0.5, 1.0, 1.5, 2.0 ugHg standards are prepared. (The lower and higher value standards are used for samples running higher or lower than those normally encountered. Stopper standards while awaiting analysis.

3. INSTRUMENTAL ANALYSIS - MAS-50A Mercury Analyzer:

Set up instrument flow system by connecting dynapump output to bubbler tube; bubbler outlet to trap (50 mL flask) side arm; drying tube containing magnesium perchlorate from mouth of trap flask to MAS-50A "in"; MAS-50A "out" to dynapump inlet. Turn MAS-50A power switch on. MAS-50A pump off. Dynapump off. Allow to warm up 1 hr. Memory off. Meter set to %T for entire run. Turn dynapump on. Leave on for duration of run.

*Start Here: Shutter open set 100%T. Shutter closed set 0%T. Shutter open check 100%T. Memory on. Remove stopper from flask of standard or sample. Add 20 mL of hydroxylamine hydrochloride/sodium chloride soln. (15 g of each dissolved separately, mixed and diluted to 1L) from dispenser. Swirl to mix. Permanganate color is destroyed. Add 10 mL of stannous chloride soln (10% W/V in 0.5n sulfuric acid) from dispenser. Immediately insert bubler into flask and insure a leakproof joint at ground stopper. Meter will swing up scale to indicate the amount of mercury present. Memory on will record the highest meter travel before it begins to drop. Meter should reach its maximum in about 2 min. Record %T (estimate to nearest 0.2%T or 1/5 of scale division). Turn memory off. Remove bubbler from flask. Rinse bubbler tube. Meter will return downscale as mercury vapor is vented out of instrument. Go to *Start Here for successive standards and samples. Run 1 ugHg standard after last sample.

4. CALCULATIONS:

Convert %T to absorbance units using conversion tables or formula A.U.=2-Log (%T). Plot of A.U. vs ugHg is linear to 1.5 ugHg and falls off slightly from 1.5 to 2 ugHg. Calculate ugHg or read from calibration plot. Divide ugHg by grams sample to get ug/g or parts per million of Hg in sample.

5. QUALITY ASSURANCE:

1. Glassware: Erlenmeyer flasks used in the analysis must be rotated in use--i.e., the same flasks should not be used for standards, blanks, etc. Labels should be erased from flasks when they are cleaned and flasks chosen at random for each set of analysis.

2. NBS RM-50: The size of the sample of albacore tuna reference material weighed out for analysis must be in the range of 0.2 to 0.25g. Smaller samples may not be homogeneous and usually result in high recovery values. Larger samples will be difficult to digest properly.

3. QA Analyses: The following analyses are run as part of the analytical quality assurance program:

Duplicate of actual sample	1 per 20 samples
Blank (flask carried through the procedure-no hydrogen peroxide)	1 per 30 samples
Reference material or spiked sample	1 per 30 samples

If the project consists of <30 samples, the above QA analyses are run at least once. Currently a procedure blank is run every few days, and RM-50 reference samples and duplicate samples are analyzed on alternate days. QC criteria: the following guidelines for Hg data acceptability are applied by the lab:

Procedure Blanks should not exceed 3x the normal blank level (limit .06 ug Hg -- about .03 AU or 93% T).

RM-50 value should fall in the range of .85 to 1.10 ppm Hg.

The difference between the results of duplicate sample analyses should not exceed 10% of the average or .05 ppm Hg, whichever is larger.

QA analyses which produce unacceptable results should be repeated in the next analytical run. Two successive QA failures indicate the need to investigate for possible problems before continuing running routine samples.

4. DOCUMENTATION:

All QA data are kept on file as part of the lab's permanent records and they are transmitted with the final project report to the appropriate personnel.

HG1102.DOC
06/22/90

Pb1.102 DETERMINATION OF LEAD IN TISSUES

NYS DEPT. ENVIRONMENTAL CONSERVATION
Hale Creek Field Station
Toxic Substances Monitoring Laboratory

REFERENCE: Analytical Chemistry 1986, 58, 2614-2617

ABSTRACT OF METHOD:

Small portions of wet or freeze-dried tissues are blended with a suspension mixture consisting of nitric acid, methanol, matrix modifier, and glycerin using a high speed homogenizer. Aliquots of this suspension are dispensed onto the platform of a graphite furnace AA using an autosampler. Drying, charring, and atomization take place within the inert atmosphere of the furnace, reducing chances for contamination of the samples as compared with the commonly used dry ashing or wet digestion techniques. Quantitation is by comparison to standards which also contain nitric acid, matrix modifier, and glycerin.

1. REAGENTS AND STANDARDS:

Reagents used for this method should all be Analytical Reagent grade, except for nitric acid which is "Ultrex" ultrapure grade. Very high quality deionized water such as "Nanopure" is used throughout the method.

A. Reagents:

1) Pb Matrix Modifier = 5% w/v aqueous solution of $NH_4H_2PO_4$.

2) Suspension Medium = 20 ml Pb Matrix Modifier + 5 ml nitric acid + 25 ml methanol + 50 ml glycerin.

B. Standards:

#1 diln. 15 ml 1000 ppm stock (Fisher Certified Atomic Absorption Standard SL21-500, or equivalent) diluted to 1000 ml (.2% v/v nitric acid) 15 ug/ml

#2 diln. 10 ml #1 diln. diluted to 200 ml (.2% v/v nitric acid) .75 ug/ml

#3 diln. 5 ml #2 diln. + 10 ml M.M. + 2.5 ml Nitric Acid + 25 ml Glycerin + water. Total Volume = 50 ml .075 ug/ml or 750 pg/10 ul Injection

2. SAMPLE PREPARATION:

Tissue samples submitted for analysis should be thoroughly ground. Wet samples should be thawed and mixed, and freeze dried samples should be pulverized and mixed prior to withdrawing a portion for analysis, in order to improve sample homogeneity and obtain a representative subsample. Weigh ca 200 mg of samples or standard reference materials into 17 x 100 mm polypropylene centrifuge tubes. Add 5 ml of suspension medium with an Oxford Macro-Set pipettor and cap prior to analysis. Procedure blanks are prepared by pipetting suspension medium into an empty centrifuge tube and processing it as if it contains tissue.

Blend sample for 60 seconds at #7 speed of a Brinkmann Polytron PT 10-35 Homogenizer with a PTA 10 ST blender head with Teflon bearing (not bronze bearing which causes contamination problems that show up in procedure blanks).

Immediately after blending, use several small portions of the suspension to rinse the 2 ml polystyrene AS-40 auto sampler cup. Fill cup ca 2/3 with sample suspension and place in AS-40 for injection into AA furnace. See the "AA Determinative Step" section for further details.

The blender must be thoroughly cleaned between samples by an initial rinsing with water from a plastic wash bottle and wiping of the blender head, followed by three repetitions of a cleaning procedure consisting of running the blender for several seconds in ca 100 ml DI water, rinsing with a washbottle and wiping with a Kimwipe. The stream from the washbottle should be directed into the openings alongside the shaft and into the blender head to dislodge tissue particles from the previous sample. The beaker used for blender cleaning must be thoroughly cleaned with glassware detergent (such as Micro) and a brush, tapwater, and DI water between blendings to avoid carryover of material from the previous cleaning step. Final drying of the blender head with Kimwipes avoids dilution of the next sample analyzed.

3. AA DETERMINATIVE STEP:

This method is based on the Stabilized Temperature Platform Furnace (STPF) concept which uses a platform tube, addition of a matrix modifier, and maximum power heating to reduce interferences to the analytical signal. In this case, peak height is used instead of area, which is often a part of STPF conditions. Set up the AA (Perkin-Elmer Model 5000) and graphite furnace (P-E HGA-500) with parameters from the attached table. Set up the autosampler (P-E AS-40) for "Method 1" analyses: AZ = suspension medium reagent blank, S1=750 pg Pb standard containing MM and glycerin, Cup #1=suspended sample. Flushing of the viscous glycerin solutions is improved by the use of a dilute surfactant solution (such as .5% v/v aqueous Igepal CO-630) in the

AS-40 rinse bottle. The data station (P-E Model 3600) directs AA output to the lineprinter via the utility command COPY COMR:, PRNT:.

Standard Operating Procedures for graphite furnace AA systems include frequent inspections and periodic cleaning of furnace windows, contact rings,and graphite tubes. Since tissue samples are actually ashed in the platform, it is necessary to periodically remove the platform from the tube and clean out the accumulated residue by careful scraping with an X-acto Knife or scalpel.

The AA is calibrated in the concentration mode to read out directly in pg Pb. Prior to running each sample, set AA PRINT # = 0, press READ until the average of the 3 replicates and coefficient of variation are printed, and press AZ to zero the instrument. Enter desired sample # as next PRINT #, place sample suspension in Cup #1 position of the AS-40, key in (1) (Manual) to select Cup #1 and press START to begin the AS-40's sample injection cycle. Triplicate analyses allow the analyst to edit the data by discarding outlying values, which may have been caused by some atypical or intermittent problem.

Calculations:

(g sample/5ml) x 2000 = ug sample /10 ul injection

$$\frac{\text{pg Pb found in 10 ul injection}}{\text{ug sample in 10 ul injection}} = \text{ppm Pb in sample}$$

Plot of standard calibration is linear to 750 pg Pb, then there is a slight fall off curvature. One experiment showed that standards and standard additions plots slopes agreed to within 5%. Using 150 mg samples, it should be possible to quantitate samples in the range of .1 to 2.5 ppm Pb.

High level samples:

A minor modification to the AA detrminative step, which reduces the sensitivity about 17x, allows the quantitation of 150 mg samples containing .4 to 40 ppm Pb. For details see Appendix I.

4. QUALITY ASSURANCE:

Analytical data quality assurance depends on a combination of factors: good equipment and operating procedures, experience with the general techniques and the specific method, and the analysis of QC samples designed to measure various aspects of data quality. Perkin-Elmer suggests that instruments receive periodic (every 1-2 years) preventive maintenance care provided by a service engineer.

It is obvious that proper procedures and techniques play a major role in producing good quality data. As the analyst gains experience with a given method, QC data should fall within closer tolerances; if it does not, this indicates that something unforeseen may be affecting the data. Experience also enables the analyst to decide if a single outlying data point can be discarded if other data indicate that the procedure is basically in control, or if that sample (or several samples) should be repeated.

A. Sample Sizes:

Large samples appear to settle out faster from suspension than smaller samples. The issue of sample homogeneity in regard to analyzing very small subsamples remains to be addressed in further detail. Intuitively, it would appear that well-ground samples of a single tissue type (e.g., mink liver) would show better homogeneity than a ground fish sample which contains muscle, skin, bones, scales, etc. Samples in the range of 150-400 mg/5 ml have been analyzed using this technique.

B. QC Analyses:

1 Procedure Blank
1 SRM
2 Sample Duplicates

are included in each batch of 16 analytical determinations (12 samples + 4 QC). The values in parentheses in the following paragraphs are those obtained in Pb projects completed between August 1988 and June 1990.

1] Procedure Blanks are a measure of contamination. Results are calculated as equivalent concentration in a 200 mg sample. (n=31, ave. = .0147)

2] Detection Limits are calculated as (approx.) 4 x Procedure Blank expressed as ppm Pb in a nominal 200 mg sample. (.06 ppm Pb)

3] Standard Reference Materials are a measure of the accuracy of the method.
SRM 1566 Oyster Tissue .48 ppm Pb True (n=26, ave. = .378, S.D. = .052, 78.8% Rec.)

4] Duplicate sample analytical results are used to calculate an estimate of standard deviation, which is a measure of the precision of the analytical method. (n=16 pairs, .035 ppm Pb)

5] Standards run as samples are a simple measure of the stability of the AA calibration. (n=46, ave. = 99.8% Rec.)

6] The instrument response for a standard is a measure of instrument sensitivity. The characteristic amount is defined as the pg Pb which produce an instrument response of 1% absorption. (7-8 pg Pb, n=28, ave. = 7.35, 6.0% RSD)

C. Quality Assurance Acceptance Criteria:

Initial QA acceptance criteria are fairly broad when an analytical method is first implemented in a laboratory. It is expected that these tolerances can be narrowed as the laboratory gains experience with the method.

1] Standards run as samples should be within 90-110% recovery. Larger differences indicate the need to recalibrate with reagent blank A2 and S1 standard.

2] Procedure blanks equivalent to greater than ca .03 ppm Pb in a nominal 200 mg sample will produce Detection Limits > .1 ppm Pb, which may not be suitable for some purposes.

3] Estimates of standard deviation calculated from duplicate analyses should be less than .1 ppm Pb for low level samples. Calculated relative percent differences may appear to be excessively high as ppm Pb levels decrease (and approach zero).

4] Characteristic amounts of about 7 pg Pb for 1% abs indicate long term instrument stability. Poorer sensitivity, indicated by larger characteristic amounts may indicate degradation of standards or graphite tube. Acceptable analytical results may be obtained on a short term basis as long as the change in sensitivity is very gradual.

5] Standard Reference Material recoveries are running somewhat below 100%. Recoveries of 65 to 120% will be deemed to be acceptable.

QA analyses which produce unacceptable results should be repeated in the next analytical run. Two successive QA failures indicate the need to investigate for possible problems before continuing running routine samples.

D. Quality Assurance Documentation:

QA/QC data should be tabulated and retained as part of the laboratory's records. Summary QA/QC data should be included as part of analytical reports sent out of the laboratory.

PB1102.DOC
06/22/90

DETERMINATION OF LEAD IN TISSUES

HGA-500 GRAPHITE FURNACE PROGRAM AND AA PARAMETERS

STEP	1 Warm	2 Dry	3 Char	4 Char	5 Atom	6 Clean	7 Cool	8	9
PARAMETERS:									
TEMP (C)	130	600	800	800	2200	2500	20		
RAMP (Sec)	1	40	3	1	0	1	1		
HOLD (Sec)	10	0	5	5	2	2	9		
READ (Sec)					0				
RECORDER (Sec)					-5	0	0		
BASELINE (Sec)					-3				
INT. AR (ml/min)	300	300	300	0	0	300	300		
TOTAL TIME (Sec) (80 seconds)	11	40	8	6	2	3	10		

Tube Type	Platform
Injection	10 ul
Runs	3 x each with coefficient of variation
Wavelength	283.3 nm
Slit	.7 nm Low
Lamp	Hollow Cathode 8 ma
AA Modes	AA-BG, CONC, PEAK HT, t = 2 sec.
AZ	Suspension Medium
S1	750 pg Pb

APPENDIX I

DETERMINATION OF LEAD IN TISSUES MODIFICATION OF METHOD Pb 1.102 FOR HIGH LEVEL SAMPLES

ABSTRACT OF MODIFICATIONS;

The sensitivity of this method can be reduced about 17x by maintaining a flow of 300 ml/min of Argon through the graphite furnace during the atomization step. This should allow quantitation of 150 mg samples in the range of about .4 to 40 ppm Pb. AA scale expansion is about 2.7, and Mo=120 pg/1% abs. The stop-flow determination in the original method, with Mo=7-8 pg/1% abs., allows quantitation of 150 mg samples in the range of .1 to 2.5 ppm Pb.

All analytical reagents and procedures are the same as in the original method except for the following modifications:

1. B. Standards:

#1 diln. 6 ml 1000 ppm stock (Fisher certified Atomic Absorption Standard SL21-500, or equivalent) diluted to 500 ml (.2% v/v nitric acid) 12 ug/ml

#2 diln. 5 ml #1 diln. + 10 ml M.M. + 2.5 ml Nitric Acid + 25 ml Glycerin + Water Total Volume = 50 ml
12000 pg or 12.00 ng/10 ul injection

3. AA Determinative Step:

S1 = 12.00 ng Pb/10 ul injection

Calibration plot shows good linearity to 12 ng Pb.

Table of Furnace and AA Parameters:
Steps #4 & 5 change Int.Ar from 0 to 300 ml/min
S1 12.00 ng Pg

4. B. QC Analyses:

The following QC data was obtained using the high level modification between October 1988 and January 1989:

1] Procedure Blank n=1 .2 ppm Pb for a 200 mg sample.
2] Detection Limit .8 ppm Pb for 200 mg sample.
3] SRM-1566 n=2, ave. - .443, 92.3% Rec.

4] Duplicates no data
5] Standards run as samples:

STD.	n	Ave.	% Recovery
750 pg	1	770	102.7
6 ng	2	5.90	98.3
12 ng	8	11.42	95.2 (6.2% RSD)

6] Instrument response n=6, average characteristic amount = 132 pg/1% absorption, 18x reduction in sensitivity.

Cd1.102 DETERMINATION OF CADMIUM IN TISSUES

NYS DEPT. ENVIRONMENTAL CONSERVATION
Hale Creek Field Station
Toxic Substances Monitoring Laboratory

REFERENCE: Analytical Chemistry 1986, 58, 2614-2617

ABSTRACT OF METHOD:

Small portions of wet or freeze-dried tissues are blended with a suspension mixture consisting of nitric acid, methanol, matrix modifier, and glycerin using a high speed homogenizer. Aliquots of this suspension are dispensed onto the platform of a graphite furnace AA using an autosampler. Drying, charring, and atomization take place within the inert atmosphere of the furnace, reducing chances for contamination of the samples as compared with the commonly used dry ashing or wet digestion techniques. Quantitation is by method of additions.

1. REAGENTS AND STANDARDS:

Reagents used for this method should all be Analytical Reagent grade, except for nitric acid which is "Ultrex" ultrapure grade. Very high quality deionized water such as "Nanopure" is used throughout the method.

A. Reagents:

1] Cd Matrix Modifier = aqueous solution of 5% w/v $NH_4H_2PO_4$ + .1% w/v $Mg(NO_3)_2$

2] Suspension Medium = 20 ml Cd Matrix Modifier + 5 ml nitric acid + 25 ml methanol + 50 ml glycerin.

B. Standards:

#1 diln. 5 ml 1000 ppm Cd stock solution (Fisher Certified Atomic Absorption Standard SC118-500, or equivalent) diluted to 500 ml (.2% v/v nitric acid) 10 ug/ml

#2 diln. 5 ml #1 diln. diluted to 500 ml (.2% v/v nitric acid) .1 ug/ml

#3 diln. 5 ml #2 diln. + 2.5 ml nitric acid + 10 ml Cd matrix modifier + 25 ml glycerin.
Total volume = 50 ml 10 ppb

#4 diln. 10 ml #2 diln. + 2.5 ml nitric acid + 10 ml Cd matrix modifier + 25 ml glycerin. Total volume = 50 ml 20 ppb

2. SAMPLE PREPARATION:

Tissue samples submitted for analysis should be thoroughly ground. Wet samples should be thawed and mixed, and freeze dried samples should be pulverized and mixed prior to withdrawing a portion for analysis, in order to improve sample homogeneity and obtain a representative subsample. Weigh ca 200 mg of samples or standard reference materials into 17 x 100 mm polypropylene centrifuge tubes. Add 5 ml of suspension medium with an Oxford Macro-Set pipettor and cap prior to analysis. Procedure blanks are prepared by pipetting suspension medium into an empty centrifuge tube and processing it as if it contains tissue.

Blend sample for 60 seconds at #7 speed of a Brinkmann Polytron PT 10-35 Homogenizer with a PTA 10 ST blender head with Teflon bearing (not bronze bearing which causes contamination problems that show up in procedure blanks).

Immediately after blending, use several small portions of the suspension to rinse the 2 ml polystyrene AS-40 auto sampler cup. Fill cup ca 2/3 with sample suspension and place in AS-40 for injection into AA furnace. See the "AA Determinative Step" section for further details.

The blender must be thoroughly cleaned between samples by an initial rinsing with water from a plastic wash bottle and wiping of the blender head, followed by three repetitions of a cleaning procedure consisting of running the blender for several seconds in ca 100 ml DI water, rinsing with a washbottle and wiping with a Kimwipe. The stream from the washbottle should be directed into the openings alongside the shaft and into the blender head to dislodge tissue particles from the previous sample. The beaker used for blender cleaning must be thoroughly cleaned with glassware detergent (such as Micro) and a brush, tapwater, and DI water between blendings to avoid carryover of material from the previous cleaning step. Final drying of the blender head with Kimwipes avoids dilution of the next sample analyzed.

3. AA DETERMINATIVE STEP:

This method is based on the Stabilized Temperature Platform Furnace (STPF) concept which uses a platform tube, addition of a matrix modifier, peak area and maximum power heating to reduce interferences to the analytical signal. Set up the AA (Perkin-Elmer Model 5000) and graphite furnace (P-E HGA-500) with parameters from the attached table. Set

up the autosampler (P-E AS-40) for "Method 1" analyses: Cup #1 = suspension medium reagent blank, Cup #2 = 10 ppb Cd standard, Cup #3 = 20 ppb Cd standard. Reagent container contains suspension medium reagent blank for the first injection or suspended sample for the second, third, and fourth injections of each sample cycle. Flushing of the viscous glycerin solutions is improved by the use of a dilute surfactant solution (such as .5% v/v aqueous Igepal CO-630) in the AS-40 rinse bottle. The data station (P-E Model 3600) runs "MADAA.BA" a PEIOS-BASIC program which collects data from the AA, calculates analytical results by the 2-spike method of additions, and prints a brief report for each sample.

Standard Operating Procedures for graphite furnace AA systems include frequent inspections and periodic cleaning of furnace windows, contact rings, and graphite tubes. Since tissue samples are actually ashed in the platform, it is necessary to periodically remove the platform from the tube and clean out the accumulated residue by careful scraping with an X-acto Knife or scalpel.

After setting up the system as described above, turn on the data station, enter time and date, place the floppy disk containing BASIC and "MADAA" into MFD0:, and type in "MADAA.BA" from the PEIOS> prompt to automatically load BASIC and load and run the program. In response to the program prompts for Metal, Spike #1, Spike #2 enter CADMIUM, 100, 200. When requested, enter sample ID# and ug sample in 10 ul injection (see Calculations sub-section below). AA Print # = 0. With suspension medium reagent blank in reagent position of the AS-40, press <1> <Manual> to select Cup #1, which also contains reagent blank. Press AS-40 Start/Stop button twice to initiate a single injection cycle. After the furnace run, the absorbance for the blank will be displayed on the AA and on the data station screen. Press the AA "AZ" button to zero the instrument. Place the sample suspension in the reagent position of the AS-40. Check that AS-40 is at Cup #1 and AA Print # = 1, then press AS-40 Start/Stop button to begin the injection cycle. The sample will be run with no Cd added, 100 pg Cd added, and 200 pg Cd added. After "MADAA" calculates and prints the results on the data station screen and on the lineprinter, pressing any key on the data station keyboard will produce a menu. To run another sample for cadmium using 100 and 200 pg Cd spikes press <S> <Return>. The other options, <R> and <E>, return to the beginning of the program to start another Run or End the program, respectively. Calculations:

(g sample/5ml) x 2000 = ug sample /10 ul injection

$$\frac{\text{pg Cd found in 10 ul injection}}{\text{ug sample in 10 ul injection}} = \text{ppm Cd in sample}$$

Plot of standard calibration is linear to 200 pg Cd, then there is a slight fall off curvature. One experiment showed that standards and standard additions plots slopes differed by 23%, necessitating use of the method of additions. Using 150 mg samples, it should be possible to quantitate samples in the range of .1 to 1.3 ppm Cd.

The Perkin-Elmer system consisting of AS-40, HGA-500, and Model 5000 AA is capable of running fully automated method of additions analyses using a single spike. Only concentration data is reported, and a variation in any one of the readings involved could produce a valid result (one without error messages) which is inaccurate.

"MADAA" (Method of ADditions AA) is a PEIOS BASIC program which was written to accept data from the communications port of the AA, calculate analytical results using the method of additions with two spikes, and print a report. This report includes absorbances of the unspiked and spiked sample, slope, intercept, correlation coefficient of the least squares linear regression line through the data points, pg of metal found, and ppm in the sample. The correlation coefficient is a measure of how well the calculated line fits the data points, and the absorbance data can be hand plotted to visually check the fit.

The following is a brief description of the program MADAA.BA:

Lines	Function
1- 90	Heading, set up I/O & Array variables.
100- 190	Obtain data for RUN from analyst: metal being determined and spike levels in pg.
200- 290	Obtain data for SAMPLE from analyst: ID# and ug sample injected. Initializes variables for new sample.
300- 370	Inputs a line of data from AA, extracts print sequence number, A.U. & AA code, and stores data in arrays.
400- 460	Examines data: If an error code is received from AA, no calculations are attempted, but data received prior to the error are printed. Returns to get more data until sequence # =3.
500- 690	Calculation of least squares linear regression and analytical results.

700- 930	Prints report to both screen and line-printer. Heading contains metal, ID#, method, date of analysis, and ug sample injected into furnace. Raw data printed includes sequence #, an explanation of what was injected (for example, Sample + 100 pg spike), absorbance, and AA Code (such as "AZ"). Linear regression data includes slope, Y-intercept, correlation coefficient, pg metal found, and ppm in sample. Reports are divided and paginated to 3 reports per page.
1000-1100	A MENU allows the analyst to go back to start a new RUN, start a new SAMPLE using the same metal and spikes, or END the program.
1200-1250	Clears screen, ejects reports from printer, closes files, and ends program.

The key to successfully running this program is to make sure that the AA sends the proper print # to the Data Station at all times:

Printer #	Run	
0	Reagent Blank + Reagent Blank	AZ
1	Sample + Reagent Blank	
2	Sample + Spike #1	
3	Sample + Spike #2	

The program will continue to accept data from the AA until it receives data with Print # = 3, at which time it begins the calculations. Any of the earlier data, with print #s 0-2, can be repeated, if desired; only the most recent data for each print # will be retained. For example, with Print # = 0, running the reagent blank produces some absorbance value. This value is printed on the screen during the run. When the analyst pressed "AZ", another line is printed on the screen (also with Print # = 0) showing A. U. = 0, and CODE = AZ. Only this second set of data (#=0, A.U.=0, Code=AZ) is saved for use in calculations and printing in the final report.

A printout of the program listing is attached.

4. QUALITY ASSURANCE:

Analytical data quality assurance depends on a combination of factors: good equipment and operating procedures, experience with the general techniques and the specific method, and the analysis of QC samples designed to measure various aspects of data quality. Perkin-Elmer

suggests that instruments receive periodic (every 1-2 years) preventive maintenance care provided by a service engineer. It is obvious that proper procedures and techniques play a major role in producing good quality data. As the analyst gains experience with a given method, QC data should fall within closer tolerances; if it does not, this indicates that something unforeseen may be affecting the data. Experience also enables the analyst to decide if a single outlying data point can be discarded if other data indicate that the procedure is basically in control, or if that sample (or several samples) should be repeated.

A. Sample Sizes:

Large samples appear to settle out faster from suspension than smaller samples. The issue of sample homogeneity in regard to analyzing very small subsamples remains to be addressed in further detail. Intuitively, it would appear that well-ground samples of a single tissue type (e.g., mink liver) would show better homogeneity than a ground fish sample which contains muscle, skin, bones, scales, etc. Samples in the range of 150-400 mg/5 ml have been analyzed using this technique.

B. QC Analyses:

1 Procedure Blank
1 SRM
2 Sample Duplicates

are included in each batch of 16 analytical determinations (12 samples + 4 QC). The values in parentheses in the following paragraphs are those obtained in Cd projects completed since June 1988.

1] Procedure Blanks are a measure of contamination. Results are calculated as equivalent concentration in a 150 mg sample. (ave. = -.005 ppm Cd, n=11)

2] Detection Limits are calculated as (approx.) 4 x absolute value of Procedure Blank expressed as ppm Cd in a nominal 150 mg sample. (:02 ppm Cd)

3] Standard Reference Materials are a measure of the accuracy of the method.
SRM 1577a Bovine Liver .44 ppm Cd True (76.8% Rec., n=16)

4] Duplicate sample analytical results are used to calculate an estimate of standard deviation, which is a measure of the precision of the analytical method. (.031 ppm Cd, 18 pairs)

5] Standards run as samples are a simple measure of the stability of the AA calibration. (96-144% Rec., n=23)

6] The instrument response for a standard is a measure of instrument sensitivity. The characteristic amount is defined as the pg Cd which produce an instrument response of .0044 absorbance-seconds. (ave. 21.0 pg Cd, n=29)

C. Quality Assurance Acceptance Criteria:

Initial QA acceptance criteria are fairly broad when an analytical method is first implemented in a laboratory. It is expected that these tolerances can be narrowed as the laboratory gains experience with the method.

1] Standards run as samples should be within 90-120% recovery. Larger differences may indicate the degradation of reagent blank and standards.

2] Procedure blanks equivalent to greater than ca .03 ppm Cd in a nominal 150 mg sample will produce Detection Limits > .1 ppm Cd, which may not be suitable for some purposes.

3] Estimates of standard deviation calculated from duplicate analyses should be less than .1 ppm Cd for low level samples. Calculated relative percent differences may appear to be excessively high as ppm Cd levels decrease (and approach zero).

4] Characteristic amounts of about 21 pg Cd for .0044 abs-sec indicate long term instrument stability. Poorer sensitivity, indicated by larger characteristic amounts may indicate degradation of standards or graphite tube. Acceptable analytical results may be obtained on a short term basis as long as the change in sensitivity is very gradual.

5] Standard Reference Material recoveries are running somewhat below 100%. Initially, recoveries of 65 to 135% will be deemed to be acceptable.

QA analyses which produce unacceptable results should be repeated in the next analytical run. Two successive QA failures indicate the need to investigate for possible problems before continuing running routine samples.

D. Quality Assurance Documentation:

QA/QC data should be tabulated and retained as part of the laboratory's records. Summary QA/QC data should be included as part of analytical reports sent out of the laboratory.

CD1102.DOC
06/22/90

DETERMINATION OF CADMIUM IN TISSUES

HGA-500 GRAPHITE FURNACE PROGRAM
AND AA PARAMETERS

STEP	1 Dry	2 Dry	3 Dry	4	5	6 Char	7 Atom	8 Clean	9 Cool
PARAMETERS:									
TEMP (C)	130	230	600	600	600	900	1800	2300	20
RAMP (Sec)	5	20	30	1	1	3	0	1	1
HOLD (Sec)	45	10	0	0	0	12	2	2	9
READ (Sec)							0		
RECORDER (Sec)							-5	0	0
BASELINE (Sec)							-3		
INT. Ar (ml/min)	300	300	300	300	300	300	300	300	300
TOTAL TIME (Sec) (142 seconds)	50	30	30	1	1	15	2	3	10

Tube Type — Platform

Injection — 10 ul sample suspension + 10 ul spiking solution

Wavelength — 228.8 nm

Slit — .7 nm Low

Lamp — Hollow Cathode 8 ma

AA Modes — AA-BG, ABS, PEAK AREA, t = 2 sec.

AZ — Suspension Medium

Record absorbances for:
Sample +
- Susp. Med. 0 pg Cd added
- Spike 1 100 pg Cd added
- Spike 2 200 pg Cd added

```
1 '"MADAA.BA"  PROGRAM FOR AA CALCULATIONS
2 '            USING METHOD OF ADDITIONS
3 'PROGRAM WRITTEN BY RALPH KARCHER  04/25/88
4 'PETOS BASIC PROGRAM TO ACCEPT OUTPUT FROM AA,
5 'CALCULATE RESULTS BY METHOD OF ADDITIONS, AND
6 '  INT A REPORT OF THE DATA AND RESULTS.
7 '
8 'IF NECESSARY TO ESCAPE, PRESS 3600 <BREAK> THEN 5000 <READ>
9 '
10 OPEN "O",#1, "PRNT:"
20 OPEN "O",#2, "SCRN:"
30 OPEN "I",#3, "COMR:"
40 CLS$ = CHR$(27) + "K"
50 DIM AU(3),CONC(3),CD$(3)
60 PRINT CLS$
90 '-------------------------------------------------------------
100 PRINT "*** DATA FOR RUN ***"
110 DA$=RIGHT$(DAT$,5) + "/" + LEFT$(DAT$,2)
120 INPUT "METAL BEING DETERMINED";METAL$
130 INPUT "SPIKE #1 (PG) = ";SPIKE1
140 INPUT "SPIKE #2 (PG) = ";SPIKE2
150 CONC(0)=0:CONC(1)=0:CONC(2)=SPIKE1:CONC(3)=SPIKE2
190 '-------------------------------------------------------------
200 PRINT CLS$;"*** DATA FOR SAMPLE ***"
210 INPUT "SAMPLE ID = ";ID$
220 INPUT "MICROGRAMS SAMPLE INJECTED = ";UGS
230 FOR N= 0 TO 3:AU(N)=0:CD$(N)="":NEXT N
240 X=0:Y=0:X2=0:Y2=0:XY=0
280 PRINT:PRINT "BEGIN RUNNING SAMPLE ";ID$
290 '-------------------------------------------------------------
300 '*** INPUT DATA FROM AA ***
310 INPUT #3,A$
320 SEQ = VAL(MID$(A$,1,4))
330 AU = VAL(MID$(A$,7,8))
340 CODE$ = RIGHT$(A$,2)
350 AU(SEQ) = AU
360 CD$(SEQ) = CODE$
370 PRINT SEQ,AU,CODE$
400 '*** EXAMINE DATA ***
410 IF CODE$ <> "ER" GOTO 450
420 PRINT "ERROR CONDITION EXISTS  ";AU;CODE$
430 PRINT #1, ID$;" AA ERROR CODE ";AU;CODE$
440 GOTO 700      'PRINT DATA FOR SAMPLE W/ERROR THEN TO MENU
450 IF SEQ <>3 GOTO 310
460 '-------------------------------------------------------------
500 '*** LEAST SQUARES LINEAR REGRESSION ***
510 FOR N=1 TO 3
520 X=X+CONC(N)
530 Y=Y+AU(N)
540 X2=X2+CONC(N)^2
550 Y2=Y2+AU(N)^2
560 XY=XY+CONC(N)*AU(N)
570 NEXT N
580 XA=X/3 : YA=Y/3      'AVERAGES
590 M=(X*Y/3-XY)/(X*X/3-X2)      'SLOPE
600 B=YA-M*XA      'Y INTERCEPT
```

```
610 R= M*SQR(X2/3-XA^2)/SQR(Y2/3-YA^2)      'CORR. COEFF.
620 SAPG = B/M      'STD. ADDNS. PG FOUND
630 PPM = SAPG/UGS
690 '-------------------------------------------------------------
700 '*** PRINT REPORT ***
71   RINT CLS$
720 FOR I=2 TO 1 STEP -1
730 PRINT #I,METAL$;" IN SAMPLE ";ID$;" BY STANDARD ADDITIONS AAS:"
740 PRINT #I,"ANALYZED: ";DA$;"     ";UGS;" MICROGRAMS SAMPLE INJECTED"
750 PRINT #I      'BLANK LINE
760 PRINT #I, "#","INJECTED","SPIKE","A.U.","CODE"
770 PRINT #I, 0,"R. BLANK +",CONC(0);" PG",AU(0),CD$(0)
780 FOR N=1 TO 3
790 PRINT #I,N,"SAMPLE +",CONC(N);" PG",AU(N),CD$(N)
800 NEXT N
805 IF CODE$ = "ER" GOTO 880
810 PRINT #I
820 PRINT #I,"LEAST SQUARES LINEAR  REGRESSION:"
830 PRINT #I,,"SLOPE = ";M
840 PRINT #I,,"Y INTERCEPT = ";B
850 PRINT #I,,"CORRELATION COEFFICIENT = ";R
860 PRINT #I,,"STD. ADDNS. PG ";METAL$;" = ";SAPG
870 PRINT #I,,"PPM ";METAL$;" FOUND IN SAMPLE ";ID$;" = ";PPM
880 PRINT #I
890 PRINT #I,STRING$(75,42)      'PRINTS *'S
895 IF CODE$ = "ER" THEN  PRINT #I,STRING$(5,10)      '5 LF'S TO PAGINATE
900 PRINT #I,STRING$(4,10)       '4 LINE FEEDS
910 NEXT I
920 PRINT "PRESS ANY KEY TO CONTINUE"
930 IF INKEY$ = "" THEN 930
10   '*** MENU ***
101  PRINT CLS$;,,"WHAT'S NEXT ?"
1020 PRINT
1030 PRINT "<R>     START NEW <R>UN"
1040 PRINT "<S>     START NEW <S>AMPLE"
1050 PRINT "<E>     <E>ND THIS ANALYSIS"
1060 PRINT:INPUT "YOUR SELECTION (R,S,E)";WN$
1070 IF WN$ = "R" GOTO 100
1080 IF WN$ = "S" GOTO 200
1090 IF WN$ = "E" GOTO 1200
1100 GOTO 1010      'IF NOT R,S, OR E
1200 '*** EXIT PROGRAM ***
1210 PRINT CLS$
1220 PRINT #1,CHR$(12);CHR$(12)      '2 FORM FEEDS TO GET PAPER OUT
1230 CLOSE #1, #2, #3
1250 END
```

OC1.104

ORGANOCHLORINE RESIDUES
NYS DEPARTMENT OF ENVIRONMENTAL CONSERVATION
Hale Creek Field Station
Toxic Substances Monitoring Laboratory

Reference: See FDA Pesticide Analytical Manual Vol. I, Sec. 211, 253

Note: All chemicals used are pesticide grade

1. Extraction:

   a. Using an analytical balance, weigh a 250 mL flat bottom boiling flask (24/40) having 2-3 teflon boiling chips.

   b. Pour ca 200 mL Hexane into boiling flask and place on cold hot plate.

   c. Place pre-extracted glass wool in Soxhlet covering the bottom and siphon tube inlet. Carefully place a known amount of sample into Soxhlet. Be sure level of sample is below top of siphon tube. Cover sample with more glass wool. If needed, add glass stoppers to Soxhlet until level with top of siphon tube. Connect Soxhlet to condenser and boiling flask.

   d. Turn on hot plate and extract overnight (ca 18 hrs.). Turn off in morning and let cool.

   e. Carefully loosen Soxhlet connections. Drain, through siphon tube, remaining Hexane into boiling flask and remove Soxhlet.

   f. Remove boiling flask from hot plate and evaporate Hexane using the Rotary Evaporator. (T=40C)

2. Cleanup:

   a. Remove boiling flask from Rotovap, dry thoroughly and weigh. Calculate weight of Hexane-extractable material (lipid).

   b. Using a 25 mL beaker, weigh out ca 0.2g of sample (lipid) on an analytical balance.

   c. Dissolve subsample with 2-5 mL petroleum ether.

   d. Place a 22 mm ID glass chromatography column with a 300 mL reservoir in a clamp. Place small wad of Hexane-extracted glass wool in bottom of column.

e. Fill column with 10 cm of activated Florisil (675C for 6 hrs. stored overnight at 130C). Tap column to eliminate channeling in the Florisil.

f. Pour 2 cm of anhydrous Na_2SO_4 (heated at 600C for 8 hrs. stored at room temperature) into column.

g. Rinse Florisil column with ca 50 ml Pet. ether into a waste beaker. Drain until Pet. ether level is just above Na2SO4 layer. Turn off stopcock. Discard eluate.

h. Place a labelled glass 250 mL Erlenmeyer flask (24/40) underneath the column.

i. Quantitatively transfer subsample onto column. Rinse beaker several times with small portions of Pet. ether. Rinse funnel, then rinse sides of glass column.

j. Allow sample to drain through column into flask at 4-5 mL per minute. Elute until solution is just above Na2SO4 layer. Turn off stopcock.

k. 2% Elution: Pour 200 mL of 2% Ethyl-Ether/Pet. Ether (v/v) solution onto column. Elute at 4-5 mL per minute. Stop flow when solvent is just above Na2SO4 layer. Remove flask.

l. 35% Elution: Pour 200 mL of 35% Ethyl-Ether/Pet. Ether (v/v) solution onto column. Elute at 4-5 min. into a second 250 mL lass Erlenmeyer flask.

m. Sample Concentration -- 2% and 35% Elutions:

1. Place 1-2 teflon boiling chips in flask.

2. Add 10 drops of keeper solution (1 mL paraffin oil in 100 mL acetone).

3. Rinse Snyder three-ball condenser with Pet. ether and place in flask.

4. Place flask with condenser on open steambath. When 2-5 mL are left, remove flask from steambath and rinse some Pet. Ether through condenser. Remove condenser and rinse off bottom into flask.

5. Finish evaporating just to dryness on Rotovap.

n. 2% Fraction: Dilute with isooctane to an appropriate concentration and stopper. Shake briefly to dissolve sample. The sample is now ready for analysis by GC1.1**.

o. 35% Fraction: Saponification--Reference: See FDA PAM Vol. I, Sec. 211.15d.

1. Reagent: dissolve 2g anhydrous AR grade KOH in 100 mL USP 95% Ethanol.

2. Add 4 mL alcoholic KOH to flask.

3. Place flask on open steam bath and let soln. reflux for 15 min. The air flow of the hood will cool neck of flask and permit condensation on inside walls of flask.

4. Remove flask from steambath and cool. Add 6 mL distilled H20.

5. Add 10.00 mL Hexane and stopper.

6. Add 10 uL of internal standard (Endolsulfan I, 8.34 ng/uL in isooctane).

7. Shake vigorously for 30 secs. Let layers separate. Make injections into the GC from clear upper layer. Analyze by GC1.2**.

3. Calibration

a. Each day the analytical balance is used, it is calibrated using its internal 100g class S weight.

b. Analytical standard solutions are prepared from primary standards (if available; generally obtained from USEPA). Stock solutions in isooctane may be stored refrigerated up to one year. Working refrigerated standards may be used up to six months. New working standards should agree to within 10% of previous standards (as determined by gas chromatography).

c. Gas Chromatographs

1. Perkin-Elmer Sigma 115 - Sample results are calculated using external standards. At the start of a run, three standards are analyzed and a linear calibration table is calculated. A stand-

ard is then run at least once every eight injections and the calibration table recalculated. The range of the calibration table is extended 20% above the high level standard and 20% below the low level standard. The correlation coefficient (r^2) is expected to be ≥ 0.95. If $r^2 \leq 0.95$, the sample will be rerun or calculated from the standard which most closely matches it in peak area.

2. Tracor 222 - Sample results are calculated using a single point external standard. A standard, then up to eight samples, and another standard are injected. The standards are averaged and the sample results calculated. If the standard peak heights differ by >15%, the samples are rerun.

4. Quality Control Samples

a. A quality control group consists of 30 samples.

b. For every 30 environmental samples, there are analyzed:
2 reagent blanks
1 reagent spike
1 standard reference material (SRM)
and 3 duplicate samples.

c. The acceptable criteria for reagent blanks are that no peak will interfere with the quantitation at a level greater than the detection limit.

d. The acceptable limits for the spikes, SRMs, and duplicates are that the calculated results will be within $\pm$ 3 standard deviations of the expected values (see attached Tables).

e. If a quality control sample falls outside the acceptable limits, the sample is examined and possibly reanalyzed. If the reanalyzed sample still falls outside the acceptable limits, all analyses are stopped until the problem is rectified. Data from the quality control group are then considered suspect and the samples should be reanalyzed. If the samples cannot be reanalyzed, the data from that quality control group is flagged.

05/22/92

GC1 GAS CHROMATOGRAPHY ANALYSIS
NYS DEPARTMENT OF ENVIRONMENTAL CONSERVATION
Hale Creek Field Station
Toxic Substances Monitoring Laboratory

GC1.103 Perkin-Elmer Sigma 115
SPB-1 Methyl Silicone bonded phase capillary column
Fused Silica 30m, 0.25 mm ID
Film Thickness: 0.25 x 10-3 mm
Flow: Approx. 2 mL/min He
Column head pressure: 26 psig at 280C
Electron Capture Detector: Range 4, make up gas 60 mL/min N2
Injector: 235C Inlet vent=on at 0.80 min
Detector: 325C Ni-63 off at 5.20 min
Oven:* 100C 2 min
15C/min to 180C hold 0 min
8C/min to 280C hold 2 min
20C/min to 300C hold 3 min
* varies, see actual printout for parameters
Plotter: ATTN 2
Method 10 in Sigma Console

GC1.202 Tracor 222
3% SP2100 on 100/120 Supelcoport
Glass column 1.8m, 2mm ID
Flow: 50 mL/min N2
Electron Capture Detector, make up gas 10 mL/min N2
Injector: 225C
Detector: 350C Ni-63
Oven: 185C Isothermal
Electrometer input 10, bucking range 5 (12.8x10-10 amps FS)
Method 25 in Perkin-Elmer LCI-100

05/22/92

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
HALE CREEK FIELD STATION

PARAMETER TABLE AND DATA QUALITY REQUIREMENTS
FOR FISH TISSUE ANALYSES

PARAMETER	Quantitation limit (ng/g)	Estimated accuracy % recovery mean	(s%)	Estimated precision (ng/g)
Mercury	50	103	(7.4)	28
% Lipid	.01%	100	(10)	.10%
PCB AR1016	20	98.8	(11.3)	41
AR1254/60	20	103	(11.3)	48
p,p DDE	2	102	(12.0)	17
p,p DDD	2	99.3	(13.3)	6
p,p DDT	2	101	(11.6)	5
Mirex	2	102	(12.2)	3
Photomirex	5	100	(15.8)	3
Oxychlordane	10	103	(18.6)	3
trans-Chlordane	5	102	(11.1)	1
cis-Chlordane	5	100	(11.0)	2
trans-Nonachlor	5	100	(10.6)	3
HCB	2	81.6	(19.9)	4
Dieldrin	5	75.8	(12.5)	4
Endrin	5	82.4	(16.2)	10*
HCH isomers	10	100	(10)*	10*
Aldrin	10	100	(10)*	10*
Heptachlor	10	100	(10)*	10*
Heptachlor Epoxide	10	100	(10)*	10*

NOTES: A minimum of 15% of the samples analyzed are quality assurance samples representing blanks, standard reference material, duplicate analyses, and spiked recoveries.

Methods: Mercury: HG1.101 (based upon Analyt. Chem. 1968. vol. 40 p. 2085-2087)
All other parameters: OC1.103 (based upon USFDA Pesticide Analyt. Manual, Vol. I, Sec. 211, 253)

The detection limit equals the quantitation limit divided by 4.
The data reported for accuracy and precision are based upon samples analyzed from 1989 through 1991. Data reported with an * should be just considered reasonable assumptions since not enough data points exist.

02/25/92
QATBL.SJJ

METHOD 10

ANALYZER CONTROL

INJ TEMP 235
DET ZONE 1,2 350 25
AUX TEMP 25
FLOW A,B 5 5
INIT OVEN TEMP TIME 110 2

TEMP	RATE	TIME
180	15.0	0
280	8.0	2
300	20.0	3

DATA PROC

STD WT,SMP WT 1.0000 1.0000 0
FACTOR,SCALE 1 0
TIMES 19.65 9.20 12.10 14.02 17.48 19.60
SENS-DET RANGE 1 4 0.00 0 0 0
UNK,AIR 1.000 0.00
TOL 0.5000 0.001 0.1
REF PK 1.000 15.88 16.30 16.12
STD NAME

RT	RF	CONC	NAME
9.38	1.136	4.4800	ALPHA HCH
9.51	4.257	16.1000	BETA HCH
9.75	2.006	7.2000	HCB
10.01	1.804	8.0400	GAMMA HCH
10.12	1.903	8.0500	DELTA HCH
10.77	19.294	75.3984	AR1016 01
11.58	18.539	93.9200	AR1016 02
11.72	19.825	68.0896	AR1016 03
12.14	18.035	58.7584	AR1016 04
12.24	19.293	44.1296	AR1016 05
12.58	18.573	61.8880	AR1016 06
12.85	19.058	83.8080	AR1016 07
13.46	0.783	8.4000	OCS
13.53	2.307	9.3200	OXYCHLORDANE
13.95	2.411	8.7000	T-CHLORDANE
14.15	8.144	19.9000	AR1254-60 01
14.34	2.893	7.9000	C-CHLORDANE
14.54	3.362	7.2500	T-NONACHLOR
14.77	3.740	15.8600	PP DDE
14.93	10.604	20.4696	AR1254-60 02
15.29	14.524	10.3600	AR1254-60 03
15.60	5.601	21.8000	PP DDD
16.12	14.216	47.7488	AR1254-60 04
16.56	4.399	20.0400	PP DDT
16.69	10.115	36.3296	AR1254-60 05
17.17	10.528	17.7296	AR1254-60 06
17.28	10.512	15.6600	AR1254-60 07
17.51	10.310	20.3696	P-MIREX CORR
17.66	12.468	16.6296	AR1254-60 08
17.76	11.007	8.4200	AR1254-60 09
17.86	13.066	8.8600	AR1254-60 10
18.31	13.590	38.1696	AR1254-60 11
18.90	13.556	14.5000	AR1254-60 12
19.07	6.778	12.2400	MIREX
19.21	14.763	9.4500	AR1254-60 13
19.34	14.828	12.7700	AR1254-60 14

EVENT CONTROL

ATTN-CHART-DELAY 2 4 0.01

TIME	DEVICE		FUNCTION NAME				
0.00	WIDTH	W	1				
0.00	NO INT	NI	0				
0.01	NO INT	NI	1				
0.80	EXT	X	6				
5.20	EXT	X	-6				
9.18	CHART	C	25				
9.19	NO INT	NI	0				
9.21	SENS	S	A,B,S,BC	500	4	0.00	0
9.67	SENS	S	A,B,S,BC	1	4	0.00	0
9.85	SENS	S	A,B,S,BC	500	4	0.00	0
11.00	ZERO	Z	2				
11.20	SENS	S	A,B,S,BC	500	4	0.00	2
11.48	SENS	S	A,B,S,BC	500	4	0.00	0
13.30	SENS	S	A,B,S,BC	1000	4	0.00	2
13.63	SENS	S	A,B,S,BC	200	4	0.00	0
13.70	SENS	S	A,B,S,BC	1	4	0.00	2
14.00	SENS	S	A,B,S,BC	200	4	0.00	0
15.30	SENS	S	A,B,S,BC	200	4	0.00	2
15.65	SENS	S	A,B,S,BC	200	4	0.00	0
16.02	SENS	S	A,B,S,BC	200	4	0.00	2
16.10	SENS	S	A,B,S,BC	200	4	0.00	0
16.18	ZERO	Z	2				
17.11	SENS	S	A,B,S,BC	1	4	0.00	0
17.30	NO INT	NI	1				
17.31	NO INT	NI	0				
18.08	NO INT	NI	1				
18.09	NO INT	NI	0				
18.10	SENS	S	A,B,S,BC	1	4	0.00	0
18.65	ZERO	Z	2				
18.66	NO INT	NI	1				
18.67	NO INT	NI	0				
20.00	CHART	C	2				

2% STANDARD
a-HCH
b-HCH
HCB
c-HCH
d-HCH
01
02
03
HEPTACHLOR
04
05
06
ALDRIN
07
OGS+HE
OXY
15
t-CHLORDANE
01
c-CHLORDANE
t-NONACHLOR
DDE
02
03
DDD
04
DDT
05
06
07
P-MIREX
08
09
10
11
12
20
MIREX
13
14

XS HEIGHT METHOD 25 35%   LAST EDITED 15:10.1 90/09/27
Save Files: Both   Calib Seq# = 2

DATA COLL	St Time	End Time	Peak Width	A Sens	B Sens	S Sens	BCode
	2.80	10.00	-5	700	40	0.00	0
QUANT	Print Tol	Factor	Units	Smp Amt			
	0.0	1.0	PPB	1.0			
PEAK IDENT	Unret Pk	A/H Rej	Unknwn RF	Comp Tol	Comp %Tol	Ref RT	Ref Tol
	0.000	0.0	0.0	0.050	0.005	5.126	0.200
PLOTTER	Plot	Chart	Attn	Offset	Plot RT	Tic Mrk	Plot Ev
	YES	4	64	20	YES	YES	YES

COMPONENTS	CompRT	Comp RF	Comp Amt	Comp Name
	4.360	2.518428E-02	7.92	ENDOSULFAN I
	5.126	2.283879E-02	6.88	DIELDRIN
	5.738	2.943260E-02	6.18	ENDRIN

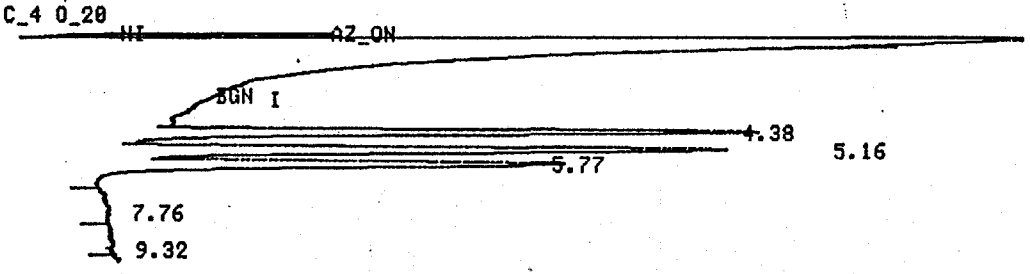
EVENTS	EvTime	Event Type	Value	Event Name
	0.000	Int Off		
	0.010	A/Z	ON	
	3.000	Int On		

Exit Setup Calib Dir Copy Delete List Edit

Exit Start Dir Del Q Del Set List Edit

FILE 456 0101-1   STARTED 10:05.3 90/10/01   L.ONT.TREND - Calib
XS METHOD 25 35%   LAST EDITED 15:10.1 90/09/27
SMP AMT   1.0

W_-5 A_64 C_4 O_20



PARAMETER TABLE AND DATA QUALITY REQUIREMENTS AND ASSESSMENTS FOR FISH TISSUE ANALYSES

Parameter	Quantitation limit (ng/g)	Estimated accuracy mean % recovery (s%)	Estimated precision s (ng/g)
Mercury	50	101.8 (5.9)	58
% Lipid	.01%	100 (10)	.10%
PCB AR1016	20	96.5 (8.8)	27
AR1254/60	20	102 (8.3)	42
p,p DDE	2	100 (9.4)	13
p,p DDD	2	98.9 (11.9)	2
p,p DDT	2	97.5 (9.0)	2
Mirex	2	102 (9.4)	4
Photomirex	5	101 (9.6)	3
Oxychlordane	5	100 (16.5)	3
trans-Chlordane	5	99.7 (9.2)	10*
cis-Chlordane	5	99.6 (8.8)	1
trans-Nonachlor	5	98.3 (10.7)	2
HCB	2	71.1 (22.7)	1
Dieldrin	5	85.8 (22.9)	4
Endrin	5	99.7 (29.3)	10*
HCH isomers	10	100 (10)*	10*
Aldrin	10	100 (10)*	10*
Heptachlor	10	100 (10)*	10*
Heptachlor Epoxide	10	100 (10)*	10*

NOTES: A minimum of 15% of the samples analyzed are quality assurance samples representing blanks, standard reference material, duplicate analyses, and spiked recoveries.

Methods: Mercury: HG1.101 (based upon Analyt. Chem. 1968. vol. 40. p. 2085-2087)

All other parameters: OC1.103 (based upon USFDA Pesticide Analytical Manual, Vol. I. Sec. 211, 253)

The detection limit equals the quantitation limit divided by 4.

The data reported for accuracy and precision are based upon samples analyzed from 1987 through 1989. Data reported with an * should be just considered reasonable assumptions since not enough data points exist.

03/01/90

I. SAMPLE RECEIPT

a) All samples received by the Lab are to be accompanied by a Collection Record and Continuity of Evidence form (see attachments I & II).

b) After comparison of samples received and the collection record, the Continuity of Evidence form is dated and signed.

c) The original forms are to remain with the lab. Copies may be returned to delivery person.

d) Depending upon sample type, the samples are to be stored under lock and key in either the cooler or freezer.

ATTACHMENT 1

FISH/WILDLIFE COLLECTION RECORD
NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF FISH AND WILDLIFE

APPENDIX I

ROM REGION_____ FOR_____ TOXIC SUBSTANCE MONITORING PROGRAM

Y COLLECTOR(S)_____ USING_____ COLLECTION METHOD.

PECIMENS PRESERVED BY_____ METHOD.

ILL IN APPROPRIATE BLANKS AS COMPLETELY AS POSSIBLE.

OR LAB SE ONLY AB ENTRY O.	COLLECTION OR TAG NO.	SPECIES	DATE TAKEN	LOCATION	AGE	SEX &/OR REPROD. CONDIT.	LENGTH ( )	WEIGHT ( )	REMARKS

322882

ATTACHMENT II

(2/84)

FISH/WILDLIFE CONTINUITY OF EVIDENCE
NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF FISH AND WILDLIFE

322883

The following items: _____

_____

_____

_____

were collected by _____

from _____ in the vicinity of _____

in the Town of _____, _____ County

on the date(s) of: _____, 19__.

Said items were in the custody of the persons listed below at all times until transferred to and hand delivered by those persons at times, dates and for purposes noted:

PRINCIPAL COLLECTOR(print name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	

FIRST RECIPIENT(print name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	

SECOND RECIPIENT(print name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	

THIRD RECIPIENT(print name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	

FOURTH RECIPIENT(print name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	

RECEIVED IN LABORATORY BY(print name)		TIME & DATE
SIGNATURE	UNIT	

LOGGED IN BY(print name)	TIME & DATE	ACCESSION NUMBERS:
SIGNATURE	UNIT	

II. SAMPLE LOGGING

a) All samples are assigned a unique serial Lab # which corresponds to a specific Tag or ID# on the sample or sample container.

b) The Lab #s are to be indicated on the Continuity of Evidence forms.

c) On the Collection Log (attachment III) the following data is transcribed:

Lab#, Tag#, Species, Location, Program, the date logged in, initials, and under the remarks column, the compound types to be determined (i.e., OC = organochlorines, Hg = Mercury, M = trace metals).

d) The original Collection Log is kept in the sample preparation lab and a copy is kept on file in supervisor's office.

322885

... ENVIRONMENTAL CONSERVATION
DIVISION OF ·SH AND WILDLIFE

LAB NO.	TAG NO.	SPECIES	LOCATION	PROGRAM	LOG IN		PROCESSING			FINAL DISPOSITION		REMARKS
										CARCASS	GROUND	
					DATE	INIT	DATE/INIT	DATE/INIT	DATE/INIT	LOC/DATE/INIT	LOC/DATE/INIT	

III. SAMPLE DISECTION

A) Samples are to be removed from freezer and allowed to partially thaw. (Large samples may be removed the previous night.)

B) At this time, a Sample Analysis Report (attachment IV) is generated and the appropriate header information is filled in (Block 1).

C) Fish: The portion of edible flesh analyzed will be referred to as the standard fillet unless otherwise noted. For some species, the procedure is modified as indicated below.

1. Procedure for Standard Filleting

a) Remove scales from fish. Do not remove the skin.

b) Make a cut along the ventral midline of the fish from the vent to the base of the jaw.

c) Make diagonal cut from base of cranium following just behind gill to the ventral side just behind pectoral fin.

d) Remove the flesh and ribcage from one-half of the fish by cutting from the cranium along the spine and dorsal rays to the caudal fin. The ribs should remain on the fillet.

e) Score the skin and homogenize the entire fillet.

2. Modificationsto Standard Fillet

Four modifications of the standard fillet procedure are designed to account for variations in fish size or known preferred preparation on methods of the fish for human consumption.

a) Some fish are too small to fillet by the above procedure. Fish less than approximately 6 inches long and rainbow smelt are analyzed by cutting the head off from behind the pectoral fin and eviscerating the fish. Ensure that the belly flap is retained on the carcass to be analyzed. When this modification is used, it should be noted when reporting analytical results.

b) Some species are generally eaten by skinning the fish. The skin from these species is also relatively difficult to homogenize in the sample. Hence, for the

following list of species, the fish is first skinned prior to filleting:

Brown bullhead	White catfish
Yellow bullhead	Channel catfish
Atlantic sturgeon	Lake sturgeon
Black bullhead	

c) American eel are analyzed by removing the head, skin and viscera; filleting is not attempted.

d) Forage fish and young-of-year fish are analyzed whole. This category is considered to be less than 150 mm (6 inches).

D) Wildlife/Other: Generally non-fish samples that are to be prepared have already been disected. See supervisor for appropriate instructions.

E) All disection tools are to be rinsed, washed with soap solution, rinsed, rinsed with distilled water and dried between each sample disection.

IV. HOMOGENIZATION

A) Depending upon sample size either the large or small food chopper is used for homogenization.

B) After the sample has been put through the food chopper, the chopper should be partially disassembled so that any remaining skin material can be removed. The skin should be cut with a knife or scissors and then combined with the rest of the sample.

C) The sample is put through the food chopper three times.

D) The sample should then be homogenized with the mixer or, if the sample is too small, by hand with a spatula.

E) The homogenized sample is then subsampled into appropriate glass bottles. Generally 2 - 10 g for metals, and 20 - 40 g for organochlorine analysis.

F) The bottles are labeled with the sample Lab # and stored in the freezer.

G) All homogenization tools are to be rinsed, washed with soap solution, rinsed, rinsed with distilled water and dried between each sample.

H) The date and initials are recorded both on the collection log and the sample analysis report.

V. FREEZE DRYING

A) If the sample is to be freeze dried, 20 - 40 g of sample are weighed into a 250 ml or 500 ml glass centrifuge bottle (block 2 of Sample Analysis Report).

B) The bottle is then stoppered (rubber stopper covered with Al foil) and placed in the freezer until frozen (generally at least 3 hrs.)

C) The refrigeration unit of the freeze dryer is turned on and after the temperature reads less than -20°C, the vacuum unit is turned on (make sure all valves are closed.)

D) After the unit has reached operating temperature (-40 to -50°C), the samples may be placed on the freeze dryer. The date and initials are recorded on the Sample Analysis Report.

E) The samples are freeze dried 24 hrs. or until the samples reach a constant weight.

F) The freeze-dried sample is weighed and then transferred to a glass screw-cap bottle, labeled with the sample Lab # and stored in the freezer.

G) Routine maintenance of the freeze dryer requires that the oil level in the vacuum pump be visually checked daily, and if under continuous use, the pump oil should be changed every 3 months. The vacuum chamber should be cleaned every two weeks and the collection chamber drained weekly.

ATTACHMENT IV

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
HALE CREEK FIELD STATION, GLOVERSVILLE, N. Y.
SAMPLE ANALYSIS REPORT

(ugg^{-1} wet wt.:dry wt.)

F # _____ SPECIES _____ SEX _____
W # _____ LOCATION _____ COLLECTION DATE _____ #1
JECT _____ MATERIAL _____

MPLE PREPARATION: BOTTLE # _____ #2
Wet + Bottle _____ Dry + Bottle _____
Bottle _____ Bottle _____
Wet Wgt. _____ Dry Wgt. _____
Date Ground _____ Date Freeze Dried _____
Bulk Sample Remaining: Yes _____ No _____

TRACTION: SOLVENT _____ DATE _____
Flask + Oil _____ Thimble + Sample _____
Flask _____ Thimble _____
Wgt. Oil _____ Wgt. Extracted _____

EAN UP: TYPE _____ DATE _____
Beaker + Oil _____
Beaker _____
Wgt. Oil _____

NALYSIS DATE: GC_____ HPLC_____ AA_____ OTHER_____
Dilutions (DF) _____

Factor = $\frac{\text{Total oil extracted x DF}}{\text{Wgt. oil analyzed x wet wgt.}}$ = _____

EMARKS: _____

TIAL REVIEW _____ DATE _____

LENGTH (cm)	
WEIGHT (g)	
% MOISTURE	
% OIL	
AROCLORS 1221	
1016	
1254	
1260	
TOTAL PCB	
p,pDDE	
p,pDDD	
p,pDDT	
TOTAL DDT	
MIREX	
HCB	
LINDANE	
TOTAL BHC	
DIELDRIN	
ENDRIN	
HEPTACHLOR EPOXIDE	
CHLORDANE	
KEPONE	
TOXAPHENE	
DEHP	
TOTAL PHTHALATES	
Hg	
Cd	

9-15-45(4/80)

322890

APPENDIX III
(UNDER DEVELOPMENT)

~~11-23-92~~
7-15-93

DATA DICTIONARY

This is to specify the standardized format of fish/wildlife contaminant data that are to be appended to the MASTER file.

FIELD NAME (MNEMONIC)	EXAMPLE/REMARKS	FIELD WIDTH	DECIMAL	TYPE
LAB NUMBER (LABNO)	123M56X2 ID number assigned by laboratory. No hyphens, spaces, etc.	8		CHARACTER
TAG NUMBER (TAGNO)	ABC123D7 ID number assigned by sampler. No hyphens, spaces, etc.	8		CHARACTER
FEATURE	LAKE ONTARIO Lake, river, wetland, site, etc, where biota were sampled	14		CHARACTER
LOCATION	HENDERSON HARBOR Geographic description of sample location	14		CHARACTER
NORTH COORDINATE (NYTMN)	12345.6 Universal ~~New York~~ Transverse Mercator coordinate (N) modified for NY State	6	1	NUMERIC
EAST COORDINATE (NYTME)	12345.6 Universal ~~New York~~ Transverse Mercator coordinate (E) modified for NY State	6	1	NUMERIC
BASIN	1234567 Numerical code for basin, sub-basin, location (BBSSLLL)	7		NUMERIC
SDATE	900628 (YYYYMMDD) Date sample(s) collected 19900628 = June, 28, 1990	8		NUMERIC
SPECIES (SPP)	LT,YP,SMB,etc. Species analyzed	5		CHARACTER
NO. ORGANISMS IN ANALYSIS (NOANLY)	48 No. in single analysis NOANLY > 1 for composite	5		NUMERIC

NOANLY = 1 for individual

FIELD NAME (MNEMONIC)	EXAMPLE/REMARKS	FIELD WIDTH	DECIMAL	TYPE
LENGTH (LENMM)	123456.7 Mean total length of fish in millimeters (mm). Length of non-fish species is defined by other than standards specified in species code data file.	7	1	NUMERIC
STANDARD DEVIATION OF LENGTH (SDLEN)	123456.7 std. deviation of total length (mm)	7	1	NUMERIC
MINIUMUM LENGTH (MINLEN)	123456.7 Total length of smallest fish in sample - units = mm	7	1	NUMERIC
MAXIMUM LENGTH (MAXLEN)	123456.7 Total length of largest fish in sample - units = mm	7	1	NUMERIC
WEIGHT (WGTG)	123456.7 Mean weight of organism in grams - units = g	7	1	NUMERIC
STANDARD DEV. OF WEIGHT (SDWGT)	1234567.8 std. deviation of total weight - units = g	8	1	NUMERIC
MINIMUM WEGHT (MINWGT)	123456.7 Weight of smallest organism in sample - units = g	7	1	NUMERIC
MAXIMUM WEIGHT (MAXWGT)	123456.7 Weight of largest organism in sample - units = g	7	1	NUMERIC
SEX	M or F Sex of organism	1		CHARACTER
AGE	12 Age of organism in years	3		NUMERIC

SAMPLE PREP (PREP)	SF, WF, KID	6		CHARACTER
I	Analyzing laboratory (CAL = Corsacha, HAZ = Hazleton, HC = Hale Creek)	3		CHARACTER

FIELD NAME (MNEMONIC)	EXAMPLE/REMARKS	FIELD WIDTH	DECIMAL	TYPE
% MOISTURE (PCTMOIST)	12.34 % moisture of sample	4	2	NUMERIC
% LIPID (PCTLPD)	12.34 % lipid content of sample	4	2	NUMERIC
AROCLOR 1016 (~~AR16~~) AR1016	1234.567 Concentration as "Aroclor 1016" units = ppm	7	3	NUMERIC
AROCLOR 1254 (~~AR54~~) AR1254	1234.567 units = ppm	7	3	NUMERIC
[illegible]CLOR 1254 & A[illegible]OCLOR 1260 (AR125460)	1234.567 units = ppm note inclusion of '12' for this mixtue of "Aroclors"	7	3	NUMERIC
AROCLOR 1260 (~~AR60~~) AR1260	1234.567 units = ppm	7	3	NUMERIC
AROCLOR 1221 (~~AR21~~) AR1221	1234.567 units = ppm	7	3	NUMERIC
AROCLOR 1248 (~~AR48~~) AR1248	1234.567 units = ppm	7	3	NUMERIC
a-HEXACHLORO CYCLOHEXANE (AHCH)	123.4567 Concentration in ppm	7	4	NUMERIC
b-HEXACHLORO CYCLOHEXANE (BHCH)	123.4567 ppm	7	4	NUMERIC
g-HEXACHLORO CYCLOHEXANE (GHCH)	1234.567 ppm also called Lindane	7	4	NUMERIC

d-HEXACHLORO CYCLOHEXANE (DHCH)	123.4567 ppm	7	4	NUMERIC

FIELD NAME (MNEMONIC)	EXAMPLE/REMARKS	FIELD WIDTH	DECIMAL	TYPE
p-p'-DDT (DDT)	123.4567 ppm	7	4	NUMERIC
ortho-para DDT (OPDDT)	123.4567	7	4	NUMERIC
p-p'-DDE (DDE)	123.4567 ppm	7	4	NUMERIC
p-p'-DDD (DDD)	123.4567 ppm	7	4	NUMERIC
ortho-para DDD (OPDDD)	123.4567 ppm	7	4	NUMERIC
cis-CHLORDANE (CISCHL)	123.4567 ppm also ALPHACHLORDANE	7	4	NUMERIC
trans-CHLORDANE (TRANSCHL)	123.4567 ppm also GAMMACHLORDANE	7	4	NUMERIC
OXYCHLORDANE (OXYCHL)	123.4567 ppm	7	4	NUMERIC
METHOXYCHLOR (MEOXYCHL)	123.4567 ppm	7	4	NUMERIC
HEPTACHLOR (HEPTACHL)	123.4567 ppm	7	4	NUMERIC
HEPTACHLOR EPOXIDE (~~HEPELEPX~~ HE)	123.4567 ppm	7	4	NUMERIC
MIREX	123.4567 ppm	7	4	NUMERIC

PHOTOMIREX (PHOMIREX)	123.4567 ppm	7	4	NUMERIC

FIELD NAME (MNEMONIC)	EXAMPLE/REMARKS	FIELD WIDTH	DECIMAL	TYPE
TOXAPHENE (TOXAPH)	123.4567 ppm	7	4	NUMERIC
TRANSNONACHLOR (TRANSNON)	123.4567 ppm	7	4	NUMERIC
OCTACHLORO-STYRENE (OCTACLST)	123.4567 ppm	7	4	NUMERIC
ENDRIN	123.4567 ppm	7	4	NUMERIC
DIELDRIN	123.4567 ppm	7	4	NUMERIC
ALDRIN	123.4567 ppm	7	4	NUMERIC
HEXACHLORO-BENZENE (HCB)	123.4567 ppm	7	4	NUMERIC
a-CHLORDENE (ACHLOR)	123.4567 ppm	7	4	NUMERIC
b-CHLORDENE (BCHLOR)	123.4567 ppm	7	4	NUMERIC
g-CHLORDENE (GCHLOR)	123.4567 ppm	7	4	NUMERIC
MERCURY (HG)	123.45 ppm	5	2	NUMERIC
LEAD (PB)	123.45 ppm	5	2	NUMERIC
CADMIUM (CD)	123.45 ppm	5	2	NUMERIC
AL PCB (TPCB)	123.4567 Total of all "Aroclors" ppm	6	3	NUMERIC

TOTAL DDT (TDDT)	123.4567 Total of DDD,DDE ,DDT OPDDD & OPDDT units = ppm	7	4	NUMERIC

FIELD NAME (MNEMONIC)	EXAMPLE/REMARKS	FIELD WIDTH	DECIMAL	TYPE
TOTAL CHLORDANE (TCHL)	Total of cis & trans chlordanes + oxychlordane + transnonaclor	7	4	NUMERIC
TOTAL HCH (THCH)	Total of AHCH,BHCH,GHCH & DHCH units = ppm	7	4	NUMERIC
TOTAL MIREX (TMIREX)	Total of mirex + photo-mirex units = ppm	7	4	NUMERIC
TOTAL DIELDRIN (TDLDRN)	Total of dieldrin + aldrin units = ppm	7	4	NUMERIC
FILENAME	Name of DEC file from which data were appended	8		CHARACTER
DATE APPENDED (DAPPEND)	Date which file was appended to MASTER file	6		DATE
VERIFIED	T = True; F = False Proofreading from collection records and lab reports.	1		CHARACTER
ARSENIC (ASN)	123.45 ppm	5	2	NUMERIC